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**(54) Lubricating oil composition**

(57) Lubricating oil compositions for use in lubricating the slideway of a machine tool or injection moulding machine.

The lubricating oil composition comprises (a) a base oil, (b) one or more friction reducing agents preferably selected from phosphoric acid esters or their alkyl ammonium salts, phosphorous acid esters and fatty acids, and (c) one or more linear alkyl amines.

Preferably, the amount of the friction reducing agent(s) (b) is 0.05 to 10.0% by weight based on the amount of the base oil (a) and the amount of the linear alkyl amine(s) (c) is 0.1 to 20.0% by weight based on the amount of the friction reducing agent(s) (b).

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## Description

The present invention relates to a lubricating oil composition, particularly for use in lubricating the slideway of a machine tool or injection moulding machine. The lubricating oil composition of the present invention is more particularly for use in a machine tool for precise machining at a low feed speed.

Positioning of a machine tool table should be controlled accurately for precise machining.

However, in the case of a machine tool having a slideway, the friction in the slideway produces elastic strain in the feed systems, reducing the accuracy of positioning.

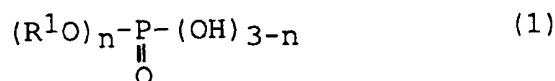
Generally, various kinds of friction reducing agents are incorporated in lubricating oil compositions for use in slideways. For example, 0.1 to 10% by weight of fat esters, fat sulphides, fatty acids and/or phosphoric esters are incorporated in a base lubricating oil.

However, conventional lubricating oil compositions have insufficient lubricity for lubricating slideways of a machine tool for precise machining. Incorporating a highly active friction reducing agent or increasing its amount was necessary for obtaining the desired lubricity. However, employing such friction reducing agent in a high concentration led to the problem of increasing the cost of such lubricating oil compositions. In addition, incorporation of a high concentration of active friction reducing agents such as fatty acids or phosphoric esters in the base oil have sometimes caused corrosion on slideways or oil supplying systems of a machine tool.

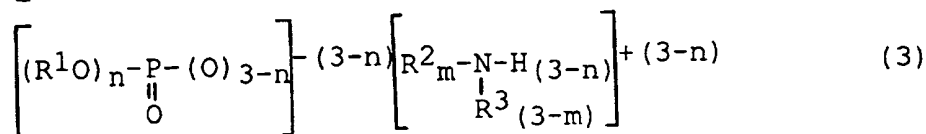
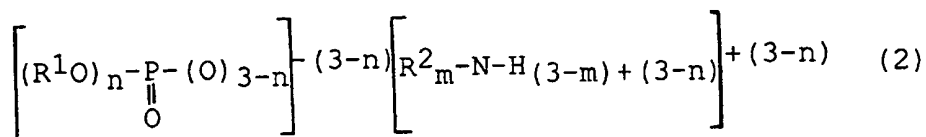
The present invention is aimed at providing a lubricating oil composition of a low cost which is particularly suitable for use in a machine tool for precise machining and improves the accuracy of positioning of sliding systems in such a tool, and which is substantially non-corrosive.

In accordance with the present invention there is provided a lubricating oil composition comprising

- (a) a base oil comprising one or more lubricating oils selected from mineral, vegetable and synthetic lubricating oils;
- (b) one or more friction reducing agents selected from phosphoric acid esters of the formula:



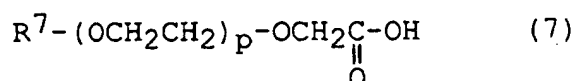
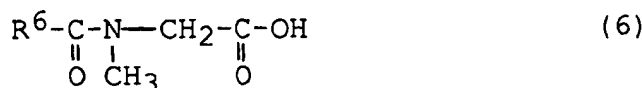
their alkyl ammonium salts of the formulae:



phosphorous acid esters of the formula:

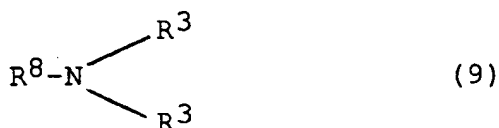


and fatty acids of the formulae:



wherein n is 1 or 2; m is 1 to 3; p is 1 to 10; R<sup>1</sup> is a C<sub>4-22</sub> alkyl, alkenyl or alkynyl group, a C<sub>6-24</sub> alkylaryl group, an alkyl group to which 1 to 10 moles of polyoxyethylene is added, or an alkylaryl group to which 1 to 10 moles of polyoxyethylene is added; R<sup>2</sup> is a C<sub>4-26</sub> alkyl, alkenyl or alkynyl group, a C<sub>6-24</sub> alkylaryl group or an aryl group; R<sup>3</sup> is a methyl or ethyl group; R<sup>4</sup> is a C<sub>8-22</sub> alkyl, alkenyl or alkynyl group or an C<sub>8-22</sub> alkylaryl group; R<sup>5</sup> is a C<sub>7-23</sub> alkyl, alkenyl or alkynyl group or a sulphurised C<sub>7-23</sub> alkyl, alkenyl or alkynyl group; R<sup>6</sup> is a C<sub>10-18</sub> alkyl, alkenyl or alkynyl group; and R<sup>7</sup> is a C<sub>8-18</sub> alkyl, alkenyl or alkynyl group; and

(c) one or more linear alkyl amines selected from the amines represented by the formulae:



wherein m and R<sup>3</sup> are as defined above; and R<sup>8</sup> is a linear C<sub>8-22</sub> alkyl, alkenyl or alkynyl group, provided that R<sup>8</sup> has more carbon atoms than R<sup>2</sup> when R<sup>2</sup> is a linear alkyl, alkenyl or alkynyl group.

The base oil (a) used in the present invention preferably has a kinematic viscosity of ISO VG 10 to 220 (at 40°C).

The mineral lubricating oil usable as the base oil (a) includes paraffinic and naphthenic mineral oils which are purified through solvent extraction and/or hydrogenation.

The vegetable lubricating oil includes rape seed oil, rice bran oil, soybean oil and others manufactured from vegetables.

The synthetic lubricating oil includes olefin oligomers, polybutenes, fatty acid esters such as esters of adipic, azelaic, phthalic, oleic or stearic acid, and polyol esters such as trimethyl propanol oleate, pentaerythritol oleate, neopentyl glycol oleate and trimethyl propanol isostearate.

These base oils can be used individually or in combination.

In the composition of the present invention, preferably R<sup>1</sup> is a C<sub>4-20</sub> alkyl group, a C<sub>16-20</sub> alkenyl group, a C<sub>4-20</sub> alkyl group to which 1 to 10 moles of polyoxyethylene is added, or a (C<sub>1-10</sub> alkyl)phenyl group to which 1 to 10 moles of polyoxyethylene is added; R<sup>2</sup> is a C<sub>4-16</sub> alkyl group or a phenyl group; R<sup>3</sup> is a methyl group; R<sup>4</sup> is a C<sub>8-14</sub> alkyl group or a C<sub>16-20</sub> alkenyl group; R<sup>5</sup> is a C<sub>8-20</sub> alkyl group, a C<sub>10-20</sub> alkenyl group or a sulphurised C<sub>16-18</sub> alkenyl group; R<sup>6</sup> is a C<sub>10-18</sub> alkyl group or a C<sub>16-20</sub> alkenyl group; R<sup>7</sup> is a C<sub>10-18</sub> alkyl group; and R<sup>8</sup> is a linear C<sub>8-20</sub> alkyl group or C<sub>16-20</sub> alkenyl group.

More preferably, the one or more friction reducing agents are selected from:

mono- or di-alkyl acid esters such as butyl acid phosphate, hexyl acid phosphate, octyl acid phosphate, 2-ethylhexyl acid phosphate, isodecyl acid phosphate, lauryl acid phosphate, stearyl acid phosphate, oleyl acid phosphate,

polyoxyethylene alkyl ether acid phosphate, and polyoxyethylene isooctylphenyl ether acid phosphate; alkyl ammonium salts of such phosphoric acid esters obtained by neutralisation of these esters with butyl amine, dibutyl amine, octyl amine, trioctyl amine, 2-ethylhexyl amine, di-2-ethylhexyl amine, t-alkylamine represented by the formula  $R^a R^b R^c CNH_2$  wherein  $R^a$ ,  $R^b$  and  $R^c$  are alkyl groups and the total number of the carbon atoms of  $R^a$ ,  $R^b$  and  $R^c$  is 3 to 25, isotridecyl amine, diisotridecyl amine, tridecyl amine, ditridecyl amine, dimethyl octyl amine or diphenyl amine;

phosphorous acid esters such as dioctyl, dilauryl and dioleoyl phosphites; and

fatty acids such as capric, lauric, myristic, palmitic, stearic, oleic, erucic, linoleic and linolenic acids or oleic acid sulfide; N-acylsarcosines such as lauroyl, myristoyl, palmitoyl and oleoyl sarcosines; and alkylether carboxylic acids such as polyoxyethylene lauryl ether carboxylic acid, polyoxyethylene stearyl ether carboxylic acid and polyoxyethylene beef tallow ether carboxylic acid.

These friction reducing agents can be used individually or in combination.

More preferably, the one or more linear alkyl amines (c) are selected from:

octyl amine, dioctyl amine, trioctyl amine, laurylamine, myristyl amine, palmityl amine, stearyl amine, oleyl amine, dioleoyl amine, distearyl amine, dimethyl stearyl amine, dimethyl oleyl amine, and dimethyl coconut amine.

The one or more friction reducing agents (b) can be used in an amount of 0.05 to 10.0% by weight, preferably 0.1 to 5.0% by weight, based on the amount of base oil (a). A lubricating oil composition containing less than 0.05% by weight of the friction reducing agent(s) (b) does not provide sufficient friction reducing effect, while a lubricating oil composition containing more than 10.0% by weight of the component (b) may corrode materials of a machine tool.

The amount of the one or more linear alkyl amines (c) is 0.1 to 20% by weight, preferably 1 to 10% by weight, based on the amount of the friction reducing agent (b). When the amount of the linear alkyl amine (c) is less than 0.1% by weight, the purpose of the present invention cannot be achieved. On the other hand, when more than 20% by weight of the linear alkyl amine (c) is incorporated, the linear alkyl amine (c) is not dissolved well in the base oil (a) when the base oil (a) is a mineral lubricating oil and the acid phosphoric ester, phosphorous ester or fatty acid used as the friction reducing agent (b) has a linear alkyl group. In addition, the activity of the friction reducing agent (b) is lowered and lubricity of the lubricating oil composition is adversely affected.

A publicly known lubricity improver can be optionally incorporated in the lubricating oil composition of the present invention. The following additives are mentioned as the examples of the lubricity improver:

oiliness improvers, extreme pressure additives and/or anti-wear additives; for example, higher alcohols, metal soaps of fatty acids, animal or vegetable fats and oils, fatty acid esters, sulphides of animal or vegetable fats and oils, ester sulphides, trialkyl phosphates, tricresyl phosphates, trialkyl phosphites, organomolybdenum friction reducing agents and zinc alkyl dithiophosphates.

In addition, antioxidants, rust inhibitors, detergents, viscosity index improvers, pour point depressants, metal deactivators, defoamers, demulsifiers and/or tackifiers, usually used as lubricating oil additives, can be optionally incorporated.

The lubricating oil composition of the present invention, wherein specific additives are combined with conventional ones, provides an excellent positioning accuracy which could not be obtained by lubricants containing a known additive individually. Therefore, machining accuracy of a machine tool can be remarkably improved by using the lubricating oil composition of the present invention. The lubricating oil composition can be also preferably used as a lubricating oil for slideways of an injection moulding machine which is required to mould materials precisely.

Therefore in accordance with the present invention there is also provided a method of lubricating the slideway(s) of a machine tool or injection moulding machine comprising lubricating said slideway(s) with a lubricating oil composition according to the present invention.

In accordance with the present invention there is further provided use of a lubricating oil composition according to the present invention for lubricating the slideway(s) of a machine tool or injection moulding machine.

In addition, the concentration of additives such as friction reducing agents can be reduced, while it was previously necessary to add conventional friction reducing agent in a high concentration for obtaining a desired lubricating performance. Therefore, the lubricating oil composition of the present invention can effectively prevent parts of a machine from corrosion caused by adding friction reducing agent in a high concentration. Further, the lubricating oil composition is inexpensive compared with conventional lubricating oil compositions.

The present invention will now be described by way of example with reference to the following working examples:

#### Examples 1 to 8 and Comparative Examples 1 to 12

#### Preparation of Sample Oil Compositions

A lubricating oil having a kinematic viscosity of ISO VG 68, a viscosity index of 110, a pour point of  $-15^{\circ}\text{C}$ , a total acid number of 0.00 mgKOH/g and a sulphur content of 0.5% by weight was used as a base oil (a).

The following compounds were used as additives:

sulfurised lard (from Dainippon Ink and Chemicals, Ltd.),

oleic acid (from Unichema International),  
 alkyl ammonium salt of phosphoric acid ester obtained by mixing octyl acid phosphate (from Asahi Denka Kogyo K.K.) and dodecyl amine (from BASF AG) (hereinafter "amine salt of phosphoric acid ester 1"),  
 alkyl ammonium salt of phosphoric acid ester obtained by mixing octyl acid phosphate (from Asahi Denka Kogyo K.K.) and di-2-ethylhexyl amine (from BASF AG) (herein after "amine salt of phosphoric acid ester 2"),  
 alkyl ammonium salt of phosphoric acid ester obtained by mixing hexyl acid phosphate (from A. & W Co.) and tertiary C<sub>12-14</sub> alkyl amine (from Rohm & Haas Company) (hereinafter "amine salt of phosphoric acid ester 3"),  
 tricresyl phosphate (from Ciba-Geigy Limited), and 2-ethylhexyl amine (from BASF AG),  
 dioleil amine (from Nippon Oil and Fats Co. Ltd.), lauryl amine (from Kao Corporation),  
 oleyl amine (from Hoechst AG), and  
 tertiary C<sub>18-22</sub> alkyl amine (from Rohm & Haas Company),  
 dilauryl hydrogen phosphite (from Sakai Chemical Industry Co. Ltd).

#### Positioning Test Procedure

A positioning test was carried out in accordance with the procedure of least input increment feed test of vertical type machining centre set forth in JIS B6338 Para. 2.8.

The machining centre was commanded through numeral control so as to feed 25 times by 1  $\mu\text{m}/\text{time}$  at intervals of 10 seconds to the plus direction on Y axis, and then, to feed to the minus direction in the same manner. Actual displacement length was measured by a laser beam length measuring instrument. The accuracy of the response of the feed of the machine table to a series of the numeral control commands was measured for the sample oil compositions of these examples and comparative examples. The feeding accuracy was evaluated by comparing backlash of least input increment, which was measured by counting the number of units necessary for shifting to the minus direction upon commanding to feed to the minus direction after commanding to feed to the plus direction. The test was carried out in an air-conditioned room at 24°C plus or minus 0.2°C. The machining centre was broken-in with the sample oil composition tested so as to lubricate the slideway surface sufficiently with the sample oil composition, and then the test was carried out 6 times. Then, the average of the results for each sample oil composition was obtained.

#### Examples and Comparative Examples

The results of Examples 1 to 6 are shown in Table 1. The results of Comparative Examples 1 to 8, 11 and 12 are shown in Table 2 and those of Examples 7 and 8 and Comparative Examples 9 and 10 are shown in Table 3.

Examples 1 to 8 relate to the lubricating oil composition of the present invention. Comparative Examples 1 to 6 and Comparative Example 9 and 10 relate to the lubricating oil compositions wherein no friction reducing agents were incorporated and/or wherein linear alkyl amines (c) of the present invention were deleted. Further, Comparative Examples 7 and 8 relate to the lubricating oil compositions wherein only specific linear alkyl amines were added. The results of the lubricating oil composition wherein branched alkyl amine was used instead of linear alkyl amine (c) were shown by Comparative Examples 11 and 12.

Comparisons of Example 1 with Comparative Example 3, Example 2 with Comparative Example 4, Example 3 with Comparative Example 5, Examples 4, 5 and 6 with Comparative Example 6, Example 7 with Comparative Example 9, and Example 8 with Comparative Example 10 indicate that lubricating oil compositions of the present invention (Examples 1 to 8) provide the machining centre with smaller backlash upon positioning and therefore, provides better positioning accuracy, compared with the lubricant oil compositions without linear alkyl amine (Comparative Examples 3 to 6, 9 and 10).

Comparison of Comparative Examples 1 and 7 and comparison of Comparative Examples 2 and 8 show that lubricant oil compositions containing the linear alkyl amine (c) only do not improve positioning accuracy of the machining centre.

Comparison of Comparative Example 6 with Comparative Examples 11 and 12 indicates that the effect of the present invention cannot be obtained by using branched alkyl amines such as 2-ethylhexyl amine and tertiary C<sub>18-22</sub> amine instead of the linear alkyl amines (c).

Table 1

|                                      |   | Example |      |      |      |      |      |
|--------------------------------------|---|---------|------|------|------|------|------|
|                                      |   | 1       | 2    | 3    | 4    | 5    | 6    |
| Amine Salt of Phosphoric Acid Ester  | 1 | -       | -    | -    | 0.75 | 0.75 | 0.75 |
|                                      | 2 | -       | 0.60 | -    | -    | -    | -    |
|                                      | 3 | -       | -    | 0.65 | -    | -    | -    |
| Oleic Acid                           |   | 0.25    | -    | -    | -    | -    | -    |
| Sulfurised Lard                      |   | -       | -    | 1.00 | -    | -    | -    |
| Dioleoyl Amine                       |   | -       | -    | -    | -    | -    | 0.01 |
| Lauryl Amine                         |   | -       | 0.01 | -    | -    | 0.01 | -    |
| Oleoyl Amine                         |   | 0.01    | -    | 0.01 | 0.01 | -    | -    |
| Backlash (number of input feed unit) |   | 3       | 7    | 7    | 3    | 6    | 6    |

Table 2

|                                      |   | Comparative Example |      |      |      |      |      |      |      |      |      |
|--------------------------------------|---|---------------------|------|------|------|------|------|------|------|------|------|
|                                      |   | 1                   | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 11   | 12   |
| Amine Salt of Phosphoric Acid Ester  | 1 | -                   | -    | -    | -    | -    | 0.75 | -    | -    | 0.75 | 0.75 |
|                                      | 2 | -                   | -    | -    | 0.60 | -    | -    | -    | -    | -    | -    |
|                                      | 3 | -                   | -    | -    | -    | 0.65 | -    | -    | -    | -    | -    |
| Oleic Acid                           |   | -                   | -    | 0.25 | -    | -    | -    | -    | -    | -    | -    |
| Sulfurised Lard                      |   | -                   | -    | -    | -    | 1.00 | -    | -    | -    | -    | -    |
| Tricresyl Phosphate                  |   | -                   | 0.50 | -    | -    | -    | -    | -    | 0.50 | -    | -    |
| 2-Ethylhexyl Amine                   |   | -                   | -    | -    | -    | -    | -    | -    | -    | -    | 0.02 |
| tert-C <sub>16-22</sub> Alkyl Amine  |   | -                   | -    | -    | -    | -    | -    | -    | -    | 0.01 | -    |
| Oleoyl Amine                         |   | -                   | -    | -    | -    | -    | -    | 0.01 | 0.01 | -    | -    |
| Backlash (number of input feed unit) |   | 12                  | 12   | 6    | 11   | 11   | 8    | 12   | 12   | 9    | 8    |

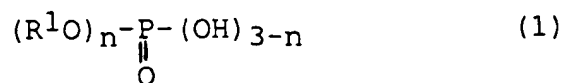
Table 3

|                                      |  | Example |      | Comparative Ex. |      |
|--------------------------------------|--|---------|------|-----------------|------|
|                                      |  | 7       | 8    | 9               | 10   |
| Hexyl Acid Phosphate                 |  | 0.50    | -    | 0.50            | 0    |
| Dilauryl Hydrogen Phosphorous Acid   |  | -       | 0.50 | -               | 0.50 |
| Oleoyl Amine                         |  | 0.01    | 0.01 | -               | -    |
| Backlash (number of input feed unit) |  | 6       | 3    | 10              | 7    |

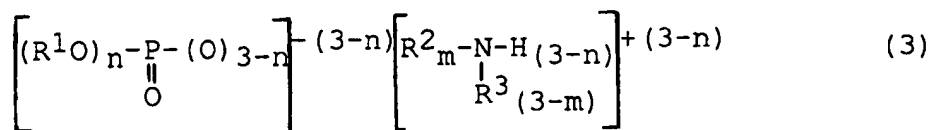
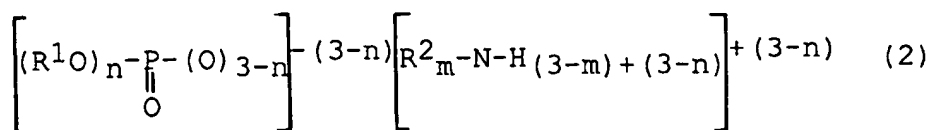
## Claims

## 1. A lubricating oil composition comprising

- (a) a base oil comprising one or more lubricating oils selected from mineral, vegetable and synthetic lubricating oils;  
 (b) one or more friction reducing agents selected from phosphoric acid esters of the formula:



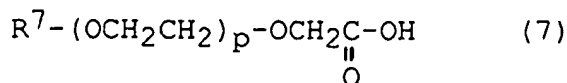
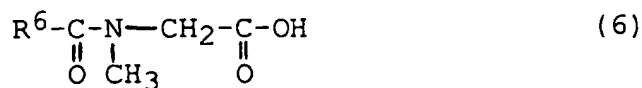
their alkyl ammonium salts of the formulae:



phosphorous acid esters of the formula:



and fatty acids of the formulae:



wherein

n is 1 or 2;

m is 1 to 3;

p is 1 to 10;

R<sup>1</sup> is a C<sub>4-22</sub> alkyl, alkenyl or alkynyl group, a C<sub>6-24</sub> alkylaryl group, an alkyl group to which 1 to 10 moles of polyoxyethylene is added, or an alkylaryl group to which 1 to 10 moles of polyoxyethylene is added;

R<sup>2</sup> is a C<sub>4-26</sub> alkyl, alkenyl or alkynyl group, a C<sub>6-24</sub> alkylaryl group or an aryl group;

R<sup>3</sup> is a methyl or ethyl group;

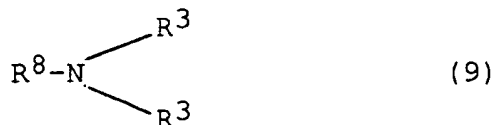
R<sup>4</sup> is a C<sub>8-22</sub> alkyl, alkenyl or alkynyl group or a C<sub>8-22</sub> alkylaryl group;

R<sup>5</sup> is a C<sub>7-23</sub> alkyl, alkenyl or alkynyl group or a sulphurised C<sub>7-23</sub> alkyl, alkenyl or alkynyl group;

R<sup>6</sup> is a C<sub>10-18</sub> alkyl, alkenyl or alkynyl group; and

R<sup>7</sup> is a C<sub>8-18</sub> alkyl, alkenyl or alkynyl group; and

(c) one or more linear alkyl amines selected from the amines represented by the formulae:



wherein

m and R<sup>3</sup> are as defined above; and

R<sup>8</sup> is a linear C<sub>8-22</sub> alkyl, alkenyl or alkynyl group, provided that R<sup>8</sup> has more carbon atoms than R<sup>2</sup> when R<sup>2</sup> is a linear alkyl, alkenyl or alkynyl group.

2. A lubricating oil composition according to claim 1 wherein

R<sup>1</sup> is a C<sub>4-20</sub> alkyl group, a C<sub>16-20</sub> alkenyl group, a C<sub>4-20</sub> alkyl group to which 1 to 10 moles of polyoxyethylene is added, or a (C<sub>1-10</sub> alkyl)phenyl group to which 1 to 10 moles of polyoxyethylene is added;

R<sup>2</sup> is a C<sub>4-16</sub> alkyl group or a phenyl group;

R<sup>3</sup> is a methyl group;

R<sup>4</sup> is a C<sub>8-14</sub> alkyl group or a C<sub>16-20</sub> alkenyl group;

R<sup>5</sup> is a C<sub>8-20</sub> alkyl group, a C<sub>10-20</sub> alkenyl group or a sulphurised C<sub>16-18</sub> alkenyl group;

R<sup>6</sup> is a C<sub>10-18</sub> alkyl group or a C<sub>16-20</sub> alkenyl group;

R<sup>7</sup> is a C<sub>10-18</sub> alkyl group; and

R<sup>8</sup> is a linear C<sub>8-20</sub> alkyl group or C<sub>16-20</sub> alkenyl group.

3. A lubricating oil composition according to claim 1 or 2 in which the one or more friction reducing agents (b) is/are present in an amount of 0.05 to 10% by weight based on the amount of base oil (a) in the composition.

4. A lubricating oil composition according to any one of claims 1 to 3 in which the one or more linear alkyl amines (c) is/are present in the amount of 0.1 to 20% by weight based on the amount of friction reducing agent(s) (b) in the composition.

5. A lubricating oil composition according to claim 4 in which the one or more linear alkyl amines (c) is/are present in the amount of 1 to 10% by weight based on the amount of friction reducing agent(s) (b) in the composition.

6. A method of lubricating the slideway(s) of a machine tool or injection moulding machine comprising lubricating said slideway(s) with a lubricating oil composition according to any one of claims 1 to 5.

7. Use of a lubricating oil composition according to any one of claims 1 to 5 for lubricating the slideway(s) of a machine tool or injection moulding machine.



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(57) Lubricating oil compositions for use in lubricating the slideway of a machine tool or injection moulding machine.

The lubricating oil composition comprises (a) a base oil, (b) one or more friction reducing agents preferably selected from phosphoric acid esters or their alkyl ammonium salts, phosphorous acid esters and fatty acids, and (c) one or more linear alkyl amines.

Preferably, the amount of the friction reducing agent(s) (b) is 0.05 to 10.0% by weight based on the amount of the base oil (a) and the amount of the linear alkyl amine(s) (c) is 0.1 to 20.0% by weight based on the amount of the friction reducing agent(s) (b).

**EP 0 711 822 A3**



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 95 20 2994

| DOCUMENTS CONSIDERED TO BE RELEVANT  |   |   |  |
|--|---|---|--|
| Category   | Citation of document with indication, where appropriate, of relevant passages   | Relevant to claim   | CLASSIFICATION OF THE APPLICATION (Int.Cl.6)   |
| X  | DATABASE WPI<br>Section Ch, Week 9403<br>Derwent Publications Ltd., London, GB;<br>Class E11, AN 94-023127<br>XP002023595<br>& JP-A-05 331 478 (TONEN CORP) , 14<br>December 1993<br>* abstract * | 1-5   | C10M133/02<br>C10M141/10<br>C10M141/06<br>//(C10M133/02,<br>133:06,<br>133:16),<br>(C10M141/06,<br>129:40,133:06,<br>133:16),<br>(C10M141/10,<br>129:40,133:06,<br>133:16,135:02,<br>137:02,137:04,<br>137:08),<br>C10N30:06,<br>C10N30:12,<br>C10N40:20 |
| X  | DATABASE WPI<br>Section Ch, Week 9114<br>Derwent Publications Ltd., London, GB;<br>Class E11, AN 91-096926<br>XP002023596<br>& JP-A-03 039 399 (TONEN CORP) , 20<br>February 1991<br>* abstract * | 1-5   |  |
| P,X  | & US-A-5 391 307 (AKIRA YAMAZAKI)<br>* column 8; example 6 *  | 1-5   |  |
| X  | EP-A-0 407 124 (TONEN CORPORATION)<br>* page 8; claims 1,14,15; examples 3-6 *  | 1-5   | TECHNICAL FIELDS<br>SEARCHED (Int.Cl.6)  |
| Y  | US-A-4 822 507 (HIDEO KANAMORI)<br>* column 3, line 40 - line 50; claims 1,4<br>*   | 6,7   | C10M   |
| Y  | DD-A-63 835 (OTTO REMETH)<br>* the whole document *   | 6,7   |  |
| A  | US-A-4 740 323 (ISOO SUZUKI)<br>* column 2, line 33 - line 40 *   | 6,7   |  |
| The present search report has been drawn up for all claims   |   |   |  |
| Place of search<br>THE HAGUE   |   | Date of completion of the search<br>27 January 1997   | Examiner<br>Hilgenga, K  |
| CATEGORY OF CITED DOCUMENTS  |   | T : theory or principle underlying the invention<br>E : earlier patent document, but published on, or<br>after the filing date<br>D : document cited in the application<br>L : document cited for other reasons<br>-----<br>& : member of the same patent family, corresponding<br>document |  |
| X : particularly relevant if taken alone<br>Y : particularly relevant if combined with another<br>document of the same category<br>A : technological background<br>O : non-written disclosure<br>P : intermediate document |   |   |  |

EPO FORM 1503 03.82 (P04C01)